UNITED STATES DEPARTMENT OF AGRICULTURE

WAR FOOD ADMINISTRATION

METHODS EMPLOYED IN THE LABORATORY ANALYSIS OF NONFAT MILK SOLIDS AND DRIED WHOLE MILK

BY THE

DAIRY AND POULTRY BRANCH

OF

OFFICE OF MARKETING SERVICES

Laboratory Located at

Room 1612 - Mallers Building
5 S. Wabash Avenue, Chicago, Ill.

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Determination of Moisture. (See Journal Dairy Science, Vol. 13, July 1930, Ind. Eng. Chem. Vol. 17, page 147, and Bulletin 911, American Dry Milk Institute, Inc. 1944). Transfer a 50-gram sample as quickly as possible to a clean. dry 300 cc. Erlenmeyer flask and immediately pour sufficient toluol into the flask to cover the sample. This requires about 75 to 100 ml. Connect the flask with the condenser by means of a distillation tube. When ready to start the distillation, fill the distillation tube with toluol by carefully pouring through the top of the condenser and bring to a boil. Shake frequently so as to prevent the sample from burning on the bottom of the flask, which would cause higher results. Just as soon as boiling has begun, reduce the heat so that the toluol will condense into the distillation tube at a rate of about four drops per second.

Approximately fifty (50) minutes after distillation has begun, dislodge any water particles in the condenser tube by means of a condenser brush and wash down with 10 ml. of toluol. Continue the distillation for an additional ten (10) minutes. Dislodge any water particles and wash down the condenser tube as before and note if there has been any increase in the moisture reading. If an increase has taken place, continue the distillation another fifteen (15) minutes or longer if necessary, then wash down as before. If no increase has taken place, the additional distillation period of fifteen (15) minutes will be unnecessary. Spray process dry milk solids does not usually require the additional distillation, but this may be necessary with roller process. The large water particles in the toluol layer in the graduated distillation tube may be dislodged readily by means of a piece of wire. The water level in the graduated distillation tube is read after it has returned to room temperature. This reading multiplied by two (2) equals the percentage of moisture in the sample.

Distillation tubes require thorough washing after each determination and condensers usually once a week. If necessary, blank determinations should be used as controls. Only moisture free toluol should be used and care should be exercised to make sure that distillation tubes are accurately calibrated.

Sediment Test. 25 grams of either spray or roller dried nonfat milk solids or 32.5 gms. of either spray or roller dried whole milk are reconstituted with 250 ml. of either filtered or distilled water and allowed to stand in a 20 ounce glass tumbler (with bottom of approximate 2" diameter) for 5 hours. The sediment in the bottom of the tumbler is compared with the American Dry Milk Institute sediment tumbler standards.

Bacterial Count. (See latest "Standard Methods for the Examination of Dairy Products" by American Public Health Association, as a guide for proper precautions, techniques and materials.) Completely dissolve 10.0 gms. of dried nonfat milk solids or 13.0 gms. of dried whole milk (weighed on a torsion balance) in 100 ml. of sterile distilled water. This may be done by weighing the dried milk on a clean, dry paper or aluminum boat and transferring the weighed samples into the dilution bottles.

The bottle is thoroughly shaken to assure homogeneous bacterial distribution. A 1:100 dilution is made from the reconstituted milk; 1 ml. of this dilution is deposited into a sterile petri dish. Approximately 10 ml. of liquefied nutrient agar (bacto-tryptone-glucose-extract-milk agar) of the proper temperature is introduced into each plate. Mix the agar and sample thoroughly and spread it evenly over the bottom of the dish by rotating the dish carefully without splashing the mixture over the edge of the plate. After the agar solidifies, invert the plates and place them in the 37°C. incubator for 48 hours.

It is essential when reconstituted milk does not meet the U.S. Grades for bacterial counts that proper dilutions be made so that the colony count is not less than 30 and not more than 300 per plate. Final plate count is expressed as the number of bacteria per cc. of the reconstituted milk.

Butterfat Determination. Butterfat is determined by the Mojonnier or modification of the Roese-Gottlieb method requiring the use of the Mojonnier extraction flask. Weigh out one gram of the well mixed dry whole milk or dry nonfat milk solids on glazed paper and transfer to a dry Mojonnier extraction flask. Add approximately 10 ml. of warm distilled water and shake until dissolved. If powder does not readily dissolve, cork and shake vigorously. Cool slightly, if necessary, to room temperature. Add three drops of 0.5% phenolphthalein solution to more clearly define the ether layer from the residue. Add 1½ ml. of strong ammonium hydroxide, mix thoroughly. Add 10 ml. of 95% ethyl alcohol, insert cork and shake thoroughly. Add 25 ml. ethyl ether, cork and shake well. Add 25 ml. petroleum ether, cork and shake well. Separate the ether layer by centrifuging, turning the handle of the Mojonnier machine 60 times at a slow rate of speed. Pour off the ether layer into a previously weighed aluminum dish. Evaporate on the hot plate at a temperature sufficient to allow complete evaporation, but not so high that spattering or vigorous boiling will result. Add 5 ml. of alcohol to the residue left in the extraction flask or tube and mix thoroughly without inserting stopper. Add 15 ml. of ethyl ether, insert cork, shake well, then add 15 ml. of petroleum ether, insert cork and again shake well. Separate the ether layer as before and pour off into the above corresponding weighed aluminum dish, containing the first extraction.

A third extraction is made (only in the case of dried whole milk) in exactly the same manner as the second, omitting the addition of alcohol. If necessary, carefully pour a few ml. of distilled water down the side of the tube just prior to pouring off the third extraction to raise the level of the aqueous layer, so ethers may be completely poured off. When only two extractions are made as with nonfat dried milk solids, the aqueous layer is raised after the second extraction. It is important that none of the aqueous layer be allowed to run into the aluminum dish.

WAR FOOD ADMINISTRATION
DAIRY AND POULTRY BRANCH

After the ether is entirely evaporated from the aluminum dish, place it in the Mojonnier oven for five minutes with the temperature at exactly 135°C. Transfer and cool to constant weight in the cooling desiccator. In weighing the dishes, both when empty and when containing the extracted fat, they should be at room temperature. This usually requires cooling in the Mojonnier desiccator for 7 to 10 minutes.

Titratable Acidity. With a pipette transfer 17.6 ml. of reconstituted dried nonfat milk solids or dried whole milk to a white evaporating dish or porcelain cup. (Reconstitution is made on basis of 10.0 gms. of dried nonfat milk solids or 13.0 gms. of dried whole milk to 100 ml. of distilled water.) Add an equal amount of distilled water and approximately 5 to 6 drops of 0.5% phenolphthalein indicator solution and titrate with N/10 sodium hydroxide until a faint pink color persists for 30 seconds. Dividing the number of ml. of alkali solution used by 20 gives the percentage of acidity as lactic acid. In order to more clearly distinguish end-points it is desirable to use a daylight lamp as a source of light.

Solubility Index. 20 grams of dried nonfat milk solids or 26.0 gms. of dried whole milk are added to 200 ml. of distilled or filtered water, (temp. of water exactly 75°F.) and agitated for 90 seconds with a Hamilton Beach electrical mixer No. 33 or a mixer giving equivalent results. Allow reconstituted sample to stand for a few minutes to permit the foam to come to the surface (this period not to exceed a total of 25 minutes from the time of mixing). Remove foam with a spoon, stir sample, and immediately pour 50 ml. in a milk solubility tube. Centrifuge tube for 5 minutes at the normal r.p.m. (1) Observe amount of insoluble material; if within the requirements for the highest U. S. grade, record the solubility index as cc. of insoluble material in the bottom of the tube. On the other hand, if this solubility index reading is above the highest grade requirement, then remove supernatant liquid to within a few cc. of the insoluble material in the bottom of the tube. Add sufficient distilled or filtered water temperature 75°F. to the 50 cc. mark on the solubility tube--shake well to loosen insoluble material. If insoluble material cannot be broken up with shaking, a twisted metal wire may be used as an aid. Centrifuge for another 5 minutes . Record the amount of insoluble material in the bottom of the tube as cc. solubility index.

1/ See table given in American Dry Milk Institute Bulletin 911, 1944, for correct speed of centrifuge.